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STATISTICAL MODELING AND OPTIMIZATION OF PROCESSING CONDITIONS OF TWIN-SCREW EXTRUDED RICE-LEGUME INSTANT BREAKFAST GRUEL

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Abstract

New product development using extrusion cooking, low cost raw materials to meet consumer preference for convenient products are increasingly becoming popular in the field of Food Engineering. The raw materials are usually complex biological materials that may undergo different transformations due to high temperature, pressure and shear in the extruder. This study investigated the effects of extrusion cooking conditions on the physical and functional characteristics of rice-cowpea breakfast gruel and modeled the relationship between the input and output variables for optimum product quality. Fifteen composite blends based on central composite rotatable design (CCRD) in a response surface methodology (RSM) were formulated and extruded at different extrusion conditions of barrel temperature, feed moisture content and rice-legume ratio in the feeds. Data generated from the analysis of the extruded products were fitted to second-order polynomial equation with optimal point located. Results showed significant ($p \leq 0.05$) variation in physical and functional characteristics of the extrudates studied. The optimal points for process conditions were obtained at 120°C barrel temperature, 20% feed moisture and 24% feed cowpea composition respectively at desirability value of 0.907, for product quality of 126.14 (expansion index), 0.214g/cm^3 (bulk density), 6.83(water absorption index), 8.46 (water solubility index), 99.02% (dispersibility) and viscosity of 12.83Nm^2. Fitted models indicated significant coefficients with adjusted R^2 of 94.70%, 88.00%, 99.70%, 99.10%, 95.90% and 83.00% respectively for expansion index, bulk density, water absorption and solubility indices, dispersibility and viscosity, while the lack-of-fit test p-values were 0.608, 0.485, 0.513, 0.505, 0.297 and 0.200 respectively. It was concluded that the fitted models could be used to adequately represent the experimental data in their natural state and when extrusion variables are carefully selected, rice-cowpea blends could produce quality gruel of optimum quality for breakfast or complimentary feeding.

Keywords: Rice, cowpea, response surface methodology (RSM), central composite design, extrusion, statistical modelling

1. Introduction

Recent advances in processing methods have provided food scientists and manufacturers with diverse innovative food processing techniques. Such methods include power ultrasound (PUS) used in the improvement of mass transfer and food preservation, pulsed electric fields (PEF) and high hydrostatic pressure (HHP) in non-thermal processing and preservation of foodstuffs, high temperature short time extrusion cooking (EC) widely used for instant breakfast cereals processing, magnetic fields (MF) and pulsed light (PL) for food preservation (Butz and Tauscher, 2002). These innovations have replaced or improved older and less efficient food processing procedures such as salting, blanching, sun drying, boiling and steaming (Brncic *et al*., 2009). EC for instance has become very important procedure during the last few decades and is expanding (Li *et al*., 2005) especially in cereal sub-sector. EC is a high temperature-short time (HTST) cooking procedure, implying that in very short processing times (1-2 min) and at high temperature, pressure and shear forces, food ingredients are being processed into foods of diverse physical, nutritional and functional qualities (Brncic *et al*., 2009).

Today, food processing machines such as extruders of different designs are being employed to produce wide range of finished products within a minimum processing time using inexpensive raw materials at reduced energy consumption. In extrusion, virtually no effluents are produced and the system is highly versatile with respect to ingredient selection and range of shapes and textures of products produced (Riaz, 2000; Souza *et al.,* 2011). Mechanically, extruders operate in dynamic steady state, where the input variables must be balanced with the outputs to be able to obtain products of desired quality. In order therefore, to obtain the required characteristics in extruded foods, the different variables of the extruder (barrel temperature, screw speed, die size to mention a few) and inputs materials (feed moisture level, composition, structure) must be set at the desired levels to give the dependent physical conditions and chemical changes within the barrel of the machine (Danbaba *et al.*, 2016, Danbaba *et al.*, 2017).

Since the ingredients used in the production of processed foods are complex biological materials, consisting of moisture, carbohydrates, protein, lipids, salts, water, vitamins, fibres and other elements which may undergo complex chemical and physical modification when subjected to high temperature, pressure and shear in the extruder barrel, these variables will ultimately affect the quality of the resulting products if not properly determined and set at the required levels. The product obtained from extrusion cooking process therefore depends on how best the various process variables are adjusted and set during the cooking process (Chessari and Sellahewa, 2001). Because of the dependence of the response variables (quality of the food) on the input variables (raw materials and machine parameters), quantitative relationship in the form of mathematical models could be developed for process and product quality optimization (Earle, 1983).

In a practical situation, the use of statistical equation to mathematically represent the relationship between independent and dependent variables that are specific to a product and process is referred to as model and have the potential of predicting real situation and provide real monetary benefits. It also has the intrinsic ability for reusability and parametric transferability (Earle, 1983; Georgiadis and Macchietto, 2000; Jun and Sastry, 2005). Modeling food process therefore allows for the exploration of product characteristics when subjected to extreme as well as intermediate process conditions without the use of actual quantity of materials, thereby saving valuable time and economic cost of operation. Since the development of highly efficient extruders for food processing, modeling the process phenomena in food system has been on the rise. This is probably because of the practical application and benefits derivable from food process models such as in development of new products, processes and for communication between people with different backgrounds (Scientists, Engineers, and Technologists). Process variables such as barrel temperature, feed moisture composition, feed ingredient composition, screw speed and size, die size have been shown to affect process and product efficiency in extrusion cooking. Moisture content for instance, during extrusion cooking provides the driving force for expansion and also contributes to the rheological properties of the melt, which in turn affect expansion. It also acts as the main plasticizer of cereal flours, which enables them to undergo glass transition during the extrusion process and thus facilitates the deformation of the matrix and its expansion.

Food extrusion process modelling has been a difficult task because of the multivariate nature of food ingredients and therefore most studies are aimed at understanding and predicting transformations taking place in the extruder and its possible effects on product qualities in specific food formulation (Ganjyal and Hanna, 2006). Falcone and Phillips, (1988), Vainionpoa *et al*. (1989),and Ganjyal *et al*. (2003) reported that the most widely used modelling approach in food system is the RSM. This is

because it allows investigator to establish mathematical relationship between input and output variables and their properties easily following a stepwise approach under minimal experimental runs. It is also an observation based approach that is more amiable to multi-component, multi-phase and multi-physics operations. It has been successfully applied for the development, improvement and optimization of biological processes where different controllable variables have individual or combined effects on the response variables (Noordin *et al.*, 2004; Krishna *et al.,* 2013; De Lima *et al*., 2010; Cheng and Frii 2010; Ponnusamy and Subramaniam, 2013).

In this study, the effects of extrusion cooking temperature (X_1), initial feed moisture content (X_2) and feed cowpea composition (X_3) on the physical and functional characteristics of instant gluten-free rice-cowpea complimentary gruels during a twin-screw extrusion cooking was studied. Statistical models to predict the relationship between the input and output variables were fitted and optimized using RSM and CCRD. The process was intended to produce breakfast cereals/complimentary gruels with high expansion at the die, good dispersibility in water, improved water absorption and protein content.

2. Theoretical framework

RSM is collection of mathematical and statistical techniques used in the optimization of the relationship between process and response variables and to determine the best possible combinations of input variables that determine a specific response. The general relationship between the independent and response variables can be therefore represented by Eqn. 1:

$$Y=f(X_i, X_2, X_3 \ldots\ldots\ldots\ldots X_k) + \varepsilon \quad (1)$$

where: Y is the response variable, $X_i, X_2, X_3 \ldots X_k$ are independent variables expressed in natural unit of measurement and ε is the error term. In RSM, it is convenient to convert the independent variables into coded variables ($x_i, x_2, x_3 \ldots x_k$) which are dimensionless with zero mean and standard deviation as it eases monitoring of experimental runs and selection of extreme values. The approximation of Y based on three or more of independent variables (Eqn. 1) is referred to as response surface methodology. Therefore, when the process variables satisfy regression analysis assumption of being measurable, continuous and controllable by experimental procedures with negligible errors, the RSM procedure can be applied through the following steps:

1. Series of trial experiments are conducted to adequately and reliably measure the acceptable lower and upper limit of the response variables of interest (Myers *et al*., 2011). Number of experimental runs required will then include the standard *2k* cube point, *2k* axial points and centre points to generate quadratic terms and replicates test at the centre point, where k is the number of independent variables considered. A CCD for three factors with 4 replicates at the centre therefore, results in a total experimental runs of $N = 2_n + (2 \times 3) + N_c$, where N is the number of runs, n is the number of independent variables being considered and Nc the number of added center points. Therefore in an experiment of three variables and six center point, there are 20 experimental runs ($N= 2^3 + (2 \times 3) + 6 = 20$ runs and a value of $\alpha = (2^n)^{1/4}$ assures rotatable design (Box and Hunter, 1975).
2. Secondly, mathematical model of the second order response surface with the best fit is developed from the experimental data to determine if there exist any significant relationship between the independent and dependent variables (Eqn. 2). Data generated from the experimental runs in the first step are subjected to regression analysis. The regression analysis models the relationship between the response and independent variables as a

mathematical function of few controllable variables and significant parameters are fitted (Montgomery, 2001; Trinh and Kang, 2010). Each response *Y* can then be represented by mathematical equation that correlates the response surface expressed as polynomial equations:

$$Y = f(y) = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i=1}^{k} \cdot \sum_{i=1}^{k} \beta_{ij} X_i X_j + \varepsilon \quad (2)$$

where: *Y* is the response variable which is related to *k* independent variables, while the parameter βj, $j=0, 1, 2 \ldots k$, are called regression coefficients which measures the expected change in y per unit change in x_j when all the remaining independent variables x_i $(I \neq j)$ are held constant.

The fitted models are validated to check if it provides an adequate approximation to the real system. Unless the model shows an adequate approximation, proceeding with the next step is likely to lead to misleading results (Myer *et al.,* 2011 and Trinh and Kang, 2010). Graphical and numerical techniques are used for the validation process (Montgomery, 2001; Trinh and Kang, 2010). The graphical method considers the nature of the residuals (difference between the experimental value and predicted values). The first plot often used is the plot of the residual versus observed order, the second is the normal probability plot and the third is the plot of predicted versus observed values. In the first plot, non-obvious pattern or uniform scattering of the residual about the zero value of the X-axis implies that the residuals are randomly distributed and renders the model acceptable for prediction purpose. In the second and third plots, an approximately straight line should be formed, and a departure would indicate a departure from a normal distribution of the residuals (Trinh and Kang, 2010). Numerical model validation is carried out by considering the *p*-value and coefficient of determination (R^2) and adjusted R^2. An R^2 and R^2_{adj} value ranges between 0 and 1. If the values are close to unity and when the two values are close to each other, the values assure a satisfactory adjustment of the fitted model.

3. Finally, the set of experimental parameters producing the optimal response value are determined through optimization by considering the coefficient of the model of each response evaluated and combination of input variable settings that optimize a single or set of response variables.

2. Materials and Methods

2.1 Materials

About 50kg of broken rice fractions from the milling of FARO 52 harvested from 2013 rain-fed production were obtained from the National Cereals Research Institute (NCRI), Badeggi, Nigeria. 10kg of brown cowpea (local variety) was purchased from the Modern market, Bida, Niger State, Nigeria. All samples of the above products were manually cleaned and packaged in a sealed polyethylene bags and stored at ambient temperature (32±2°C) until required for the conduction of experiment.

2.2 Cowpea and rice flour preparation

Cowpea flour was prepared by steeping 10kg of the brown beans in clean tap water for 30min at room temperature (32±2°C). The beans were dehulled by pounding in wooden mortar and pestle followed by several washing, and drying to about 10±2% moisture content. Dried cotyledons were then milled in an attrition mill (Locally fabricated) and sieved in a fine (150µm) laboratory sieve (Brabender OHG Duisburg). 50kg broken rice fractions were ground in the same mill and sieved in

fine (150µm) laboratory sieve (Brabender OHG Duisburg). Samples were then packaged and sealed in polyethylene bags at room temperature (32±2ºC) until required for use in carrying out test.

2.3 Ingredients formulation and moisture adjustment

Fifteen (15) formulations were prepared to contain 8 - 24% cowpea They were conditioned to appropriate moisture content (15 – 25%) by spraying with a calculated amount of water (Eqn. 3) and mixing continuously at medium speed (50 rpm) in a blender (Laboratory Scale Hobart Mixer, Hobart Corporation, Troy, Ohio, USA). The formulations were then allowed to stand for 3h to equilibrate at room temperature prior to extrusion operation to allow for the moisture level to uniformly adjust (Ascheri, 2010).

$$\text{Amount of water to be added (g)} = \frac{(M_f - M_i)\, S_w}{100 - M_f} \qquad (3)$$

where: M_f = Final moisture content (%), M_i = Initial moisture content (%) and S_w = Sample weight (g)

2.3 Extrusion cooking processing

The extrusion cooking was performed using a co-rotating twin-screw extruder (SLG 65 Twin-Screw Extruder, Jinan Saibainuo Techn. Develop. Co. Ltd, China), schematically presented in Figure 1 having operating conditions outlined in Table 1. Using the control panel, the motor speed was adjusted to give screw speed of 300 rpm and temperature adjusted to the required level. Formulations were individually introduced into the extruder barrel through the feed hopper at the rate of 30kg/h. The extrudate is allowed to follow until when the extrudate flow was steady, samples were collected and packaged until required for analysis.

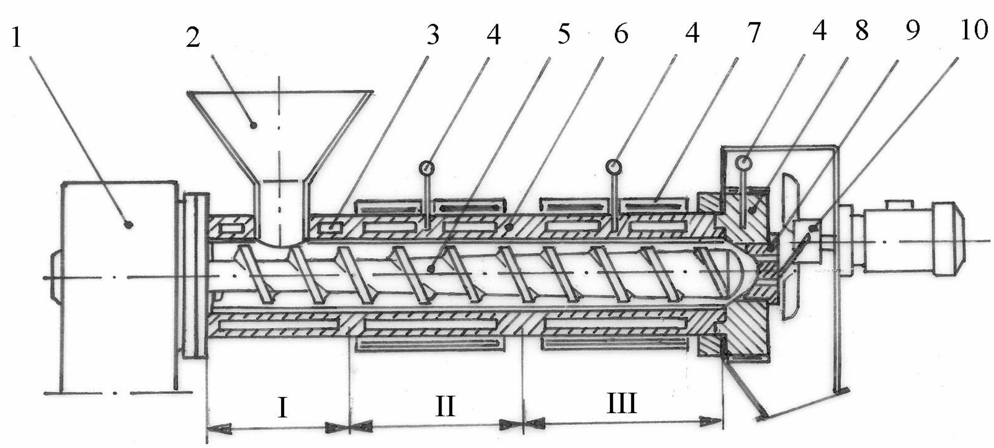

Figure 1: Schematic cross-section of extruder: 1 - motor, 2 - hopper, 3 - cooling jacket, 4 - thermocouple, 5 – screw, 6 - barrel, 7 - heating jacket, 8 - head, 9 - net, 10 -cutter, I - transport section, II – compression section, III – melting and plasticization section

Table 1: Extruder operating parameters adopted for the study

Extrusion parameters	Values
Power	16 horse power
Operating screw speed	300 rpm
Length to diameter ratio of barrel	20:1
Screw diameter	30 mm
Feed rate	30kg/h
Barrel temperature	100-140°C
Feed moisture composition	15-25g/100g
Feed cowpea composition	8-24g/100g

2.4 Experiment Design

RSM in three-factors (X_1, X_2, X_3), five-level (+α, +1, 0, -1, -α) CCRD was used to investigate the effect of the independent variables (Myer and Montgomery, 2002) (barrel temperature, feed initial moisture content, and feed cowpea composition) on the responses (expansion index, bulk density, water absorption and solubility indices, dispersibility and viscosity). Design ranges were established after several preliminary runs and were coded to lie at ±1α for the factorial points, 0 for the centre points and ±1α for axial points. The coded variables were calculated as a function of the range of interest of each natural variable as presented in Table 2, while Table 3 is the experimental matrix indicating the 15 experimental runs. The experiments were randomized to maximize the effects of unexplained variability in the observed responses due to extraneous factors, while five replicates at the centre of the design were used to allow for estimation of pure error sum of square and lack-of-fit. Two ways analysis of variance (ANOVA) at 95% level of confidence was conducted on experimental data to determine the difference among the mean treatment combinations and repeated measurements. The deviations (D) of the experimental values (OBS) from the predicted values (PRED) for the responses at the optimal condition were calculated by Eqn. 4.

$$\text{Deviation (D)} = Y - \check{Y} \qquad (4)$$

where: Y is the experimental data and $\check{Y}$ is the response predicted by the model. The empirical model representing the relationship between the input and output variables are presented in Figure 2. Assuming that the process is in a steady state and no accumulation or loss is recorded in the system.

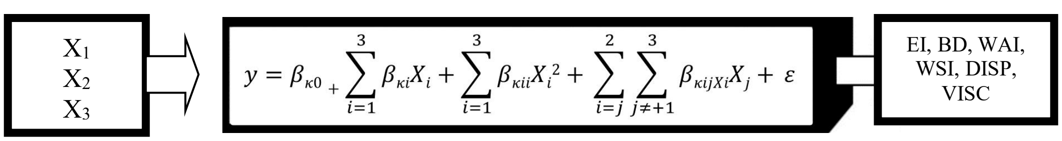

Figure 2: Empirical model representing the interaction between processing and response variables

Table 2: Independent variables and natural levels used for Central Composite Rotatable Design

Independent variables	Levels of coded variables				
	-α	Low	Medium	High	+α
	-1.68	-1	0	1	+1.68
Barrel Temperature (X_1)	86.36	100	120	140	153.64
Feed Moisture content(X_2)	11.59	15	20	25	28.41
Feed Composition (X_3)	2.55	8	16	24	29.45

Level of each variable was established based on a preliminary extrusion. The distance of the axial points from the centre point was ± 1.68, and calculated from Equation $\alpha = (2^n)^{1/4}$ where n is the number of variables.

Since interactions between the independent variables are to be considered together with linear effects, a second order polynomial regression equation (Eqn. 5) was modelled using the experimental data and optimum parameters defined using process optimizer of Minitab statistical software version 16 From the resulting values, for each of the response variable, the coefficients of the polynomial equation (β_o, β_i and β_{ij}) were determined and the equation simplified based on the influence of the factors on the final response.

$$\mathrm{Y} = f(y) = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i=1}^{k} \cdot \sum_{i=1}^{k} \beta_{ij} X_i X_j + \varepsilon \tag{5}$$

where: Y is the predicted response (either expansion index, dispersibility, water absorption and solubility indices, protein content) used as the dependent variable, k is the number of independent variables considered in the experiment; β_o constant coefficient and β_i, β_{ij} and β_{ii} are the coefficient of linear, interaction and square terms respectively, while ε is the random error term. Multivariate regression analysis was carried out on the data to yield Eqn. 6 which was used to optimize the product responses.

$$\mathrm{Y} = \beta_o + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{11} X^2{}_1 + \beta_{22} X^2{}_2 + \beta_{33} X^2{}_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \varepsilon \tag{6}$$

The coefficients with one factor (X_1, X_2, X_3) represent the linear effects of the particular factor alone, while coefficients with two of the factors (X_1X_2, X_1X_3, X_2X_3) and those with square (X_1^2, X_2^2, X_3^2) represents interaction and quadratic effects respectively.

Table 3: Outline of experimental design with variables in their coded and un-coded forms

Run No	Coded variables			Independent variables		
	X_1	X_2	X_3	X_1 (°C)	X_2 (%)	X_3 (%)
1	-1	-1	-1	100	15	8
2	1	-1	-1	140	15	8
3	-1	1	-1	100	25	8
4	1	1	-1	140	25	8
5	-1	-1	1	100	15	24
6	1	-1	1	140	15	24
7	-1	1	1	100	25	24
8	1	1	1	140	25	24
9	-1.68	0	0	86.4	20	16
10	1.68	0	0	153.6	20	16
11	0	-1.68	0	120	11.6	16
12	0	1.68	0	120	28.4	16
13	0	0	-1.68	120	20	2.6
14	0	0	1.68	120	20	29.5
15	0	0	0	120	20	16

X_1 = Barrel temperature, X_2 = Feed moisture content, X_3 = Feed cowpea composition. Duplicate runs were carried out at all design point except at the centre point (run 15) where five measurements were carried out and average recorded. The experimental runs were randomized.

To check if the fitted models provide an adequate approximation to the real system relationship, both numerical and graphical validations procedures were conducted on the fitted models. Numerical methods involve the analysis of the coefficient of determination (R^2) and adjusted coefficient of determination ($R^2{}_{adj}$) (Eqns. 7 and 8):

$$R^2 = 1 - \frac{\mathrm{SSR}}{\mathrm{SSM} + \mathrm{SSR}} \tag{7}$$

$$R^2_{adj.} = 1 - \frac{n-1}{n-p}\,(1 - R^2) \tag{8}$$

where: *SSR* is the sum of squares residual, *SSM* is the sum of squares model, *n* is the number of experiments and *p* is the number of predictors in the model not counting the constant term. In this study, R^2 value close to unity and R^2_{adj} close to R^2 were adopted as satisfactory fitting of the model to the real system, while probability value (p-value) was also used to check for the significance of each factor and interaction between the factors. The smaller the *p*-value ($p \leq 0.05$), the more significant is the corresponding coefficients. During the laboratory evaluation of the response variables, repeated measurements were taken, therefore, lack-of-fit test which shows the significance of replicate error as compared to the model dependent error was also conducted. The p-value ($p \leq 0.05$) was then used to measure whether the lack-of-fit is statistically significant or otherwise at the described level of probability. Non-significant lack-of-fit was considered desirable.

2.5 Physical Characteristics of Extrudates

Physical surface structure of the extruded samples was determined according to Barrette and Ross (1990) with slight modifications. Two samples produced at the same moisture content were selected and approximately 1.0 mm thick cross sectional was obtained and image taken with a high resolution camera (Nikon D40) positioned at 7cm above the object. Light was provided by fluorescent positioned towards the slice. Image taken was captured on a computer for physical structure analysis using photoshop software.

2.6 Moisture Determination

Moisture was determined by oven drying method. 1.5 g of well-mixed pulverized extrudate sample was accurately weighed in clean, dried crucible (W_1). The crucible containing the sample was oven-dried at 105°C for 6 h at which constant weight was obtained. Then the crucible was removed and placed in desiccator for 30 min to cool. After cooling, the sample was weighed again (W_2). The percent moisture content was then calculated by following formula (AOAC, 1984).

$$\%\,MC\ (db) = \frac{(\mathrm{W1} - \mathrm{W2})}{\text{Weight of sample}}\ x\ 100 \tag{9}$$

2.7 Determination of Expansion index (EI)

Extruded samples were cut perpendicular to the direction of extrusion using sharp blade into 10cm long pieces and measured the diameter of each piece in 5 different places using Vernier calliper. The weight of sample was measured using an electronic balance reading to 0.000g precision. Data generated for length, diameter and weight was collected from 30 pieces. From these measurements, EI was calculated by dividing the cross sectional area extrudate (A_e) by the cross sectional area (A_d)of the extruder die opening as (Alvarez-Martinez *et al.,* 1988).

$$Expansion\ index = \left(\frac{Ae}{Ad}\right)^2 \tag{10}$$

2.8 Water absorption index (WAI) and Solubility Index (WSI)

WAI and WSI were determined according to Anderson *et al.* (1969) and Jin *et al.* (1995) as modified by Onwulata *et al.* (1998). Extrudate samples were ground and sifted through a 210µm sieve and 1.0g taken and placed in a centrifuge tube. 10ml distilled water added and mixed thoroughly. After standing for 15min and shaking every 5min interval, the samples were centrifuged for 15min at

1000rpm. The supernatant was decanted and the weight gain in the gel was taken. WAI was calculated as the ratio of the weight gain to the dry gel weight. The supernatant was dried overnight at 90°C and WSI determined as weight of dried supernatant divided by the weight of dry sample, multiplied by 100 (Eqns. 11 and 12).

$$WAI\ (g) = \frac{W_S}{W_d} \qquad (11)$$

$$WSI\ (g) = \frac{W_{ds}}{W_d} \qquad (12)$$

where: W_s is weight of sediments (g), W_d is weight of dry solids (g), and W_{ds} is weight of dissolved solids in supernatant in gram.

2.9 Bulk density (BD) determination

A 5cm long cylindrical section of the extruded sample was cut using razor blade and weighed. The diameter was measured also using digital Vernier calliper. The bulk density was then calculated as the ratio of the weight of the extrudate to the calculated volume (Barret and Rose (1990).

$$BD\ (g/cm^3) = \frac{4 x W}{\pi\ x\ D^2\ x\ L} \qquad (13)$$

where: W = Weight of extrudate (g); D = Diameter of extrudate (mm); L = Length of extrudate (mm). Average of 10 readings was taken.

2.10 Viscosity Determination (RV).

Viscosity was determined using Haake Roto Visco RV1 viscometer equipped with concentric cylinders and viscosity measurement made at 30°C. The apparent viscosity of the extrudate is then measured over a range of shear rates (s^{-1}) and the relative viscosity of the solution at a given shear rate was calculated as follows: (Attia *et al*., 1979).

$$RV\ (\eta r) = \frac{\eta g}{\eta s} \qquad (14)$$

where: ŋg = Apparent viscosity of solution (Nm/S^2); Ŋs = apparent viscosity of solvent (Nm/S^2)

2.11 Dispersibility measurement

Dispersibility was determined by placing 15g of pulverized extruded sample in a 100 ml measuring cylinder and adding water to make up to 100 ml. The mixture was, then stirred for 1min 30 sec and allowed to settle for 15min. The volume of the settled particle on the cylinder was subtracted from 100 and the difference reported as dispersibility in percentage (Charunuch *et al*., 2008).

2.12 Statistical Analysis

The experiments were conducted in triplicates. Data obtained were analysed statistically using the Minitab 16 software. Two-way analysis of variance was applied to determine variability in the responses and significance of differences was verified based on the Fishers Least Square Difference (LSD) test ($p \leq 0.05$).

3. Results and Discussion

3.1 Physical characteristics of extrudates

The effect of different extrusion conditions on the physical appearance of the extruded rice-cowpea blends are presented in Figures 3 and 4. It is evident from these Figures that extrusion variables significantly affects physical surface appearance in terms of smoothness, size and grove formation (Figure 3). Similar observations were reported by Stanley and Baker (2002) and Filli *et al.* (2013). Examining the extruded samples through 2D slide indicated that starch-based extrudates have a honeycomb cellular structure. The size and number of the pores observed under the photographic enlarger varies between different conditions (Figure 4). The observation is in line with earlier reports by Faubion and Hoseney, 1982 and Lai *et al*. (1989) who observed that the size and number of cells formed in a starch extrudates reduces with increased temperature above 120^{o}C and moisture content of feed materials below 12% and the composition of raw material especially the fat and protein present.

The implication of this observation is that the mechanical strength in terms of breaking and crispiness may decrease as more pores of large sizes are formed and therefore may require system optimization to attain acceptable quality. Aguilera (2005) and Aguilera *et al*. (2007) asserted that the air or void spaces contributes significantly to the crispness of extruded puffed products. Variation in initial feed moisture content and barrel temperature are known to affect the degree of starch transformation during extrusion (Karkle, *et al.,* 2012), leading to the differences in void spaces observed in this study.

Rizvi and Mulvaney (1992) and Lee *et al* (1999) suggested that careful selection of process variables may produce extrudates with smooth outer surface and uniform air spaces. Uniform pore spaces have the advantage of decreasing water absorption rate when consumed, thereby increasing bowl life and possibly retarding staling of the product when used as breakfast cereals (Aguilera, 2005; Aguilera *et al*., 2007). Samples J and K corresponding to 154^{o}C barrel temperature, 20% moisture and 16% feed cowpea level (J) and 120^{o}C barrel temperature, 12% feed moisture and 16% cowpea (Fig. 3 and 4) may produce products that are satisfactory for gruels used as breakfast because of their smooth outer surface and uniform air space.

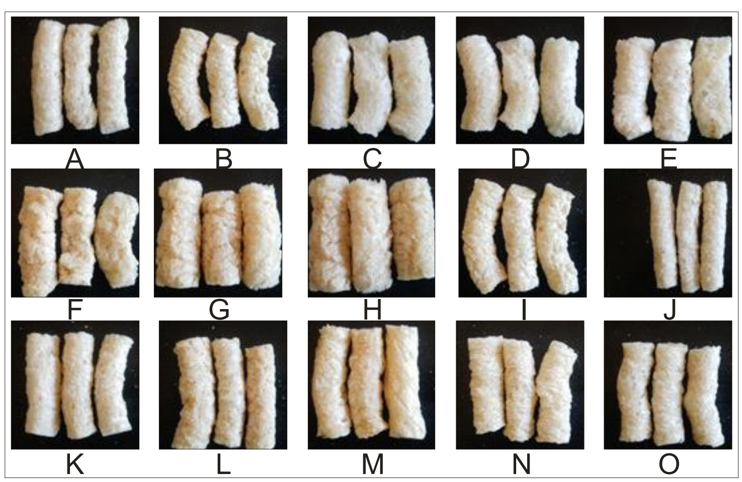

Figure 3: Photographic images of the physical state of rice-cowpea extruded foods as affected by different processing variables

Key
(A=100°C X_1, 15% X_2, 8% X_3), (B=140°C X_1, 15% X_2, 8% X_3), (C=100°C X_1, 25% X_2, 8% X_3), (D=140°C X_1, 25% X_2, 8% X_3) (E=100°C X_1, 15% X_2, 24% X_3), (F=140°C X_1, 15% X_2, 24% X_3), (G=100°C X_1, 25% X_2, 24% X_3), (H=140°C X_1, 25% X_2, 24% X_3), (I=86.5°C X_1, 20% X_2, 16% X_3), (J=153.6°C X_1, 20% X_2, 16% X_3), (K=120°C X_1, 11.6% X_2, 16% X_3) (L=120°C X_1, 28.4% X_2, 16% X_3), (M=120°C X_1, 20% X_2, 2.55% X_3) (N=120°C X_1, 20% X_2, 24.5% X_3) (O=120°C X_1, 20% X_2, 16% X_3), X_1 = Barrel temperature, X_2 = Feed moisture content, X_3 = Amount of cowpea added to rice.

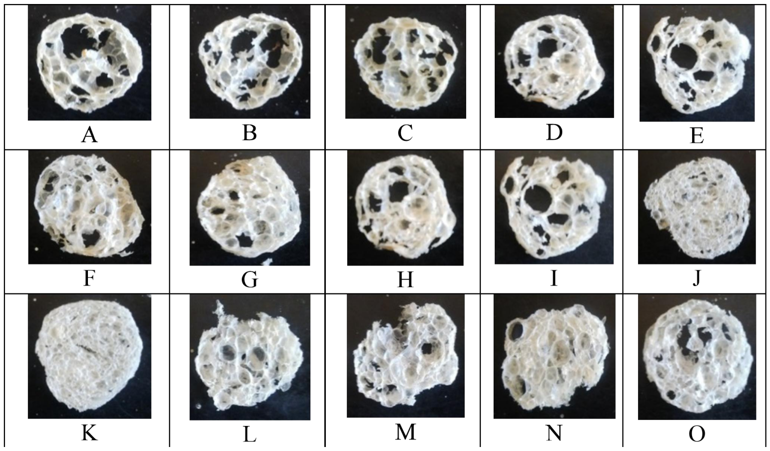

Figure 4: Photographic images of 2-D slices of rice-cowpea extruded foods showing microstructure of the samples as affected by processing variables

Key Plates 1A-1O (A=100°C X_1, 15% X_2, 8% X_3), (B=140°C X_1, 15% X_2, 8% X_3), (C=100°C X_1, 25% X_2, 8% X_3), (D=140°C X_1, 25% X_2, 8% X_3) (E=100°C X_1, 15% X_2, 24% X_3), (F=140°C X_1, 15% X_2, 24% X_3), (G=100°C X_1, 25% X_2, 24% X_3), (H=140°C X_1, 25% X_2, 24% X_3), (I=86.5°C X_1, 20% X_2, 16% X_3), (J=153.6°C X_1, 20% X_2, 16% X_3), (K=120°C X_1, 11.6% X_2, 16% X_3) (L=120°C X_1, 28.4% X_2, 16% X_3), (M=120°C X_1, 20% X_2, 2.55% X_3) (N=120°C X_1, 20% X_2, 24.5% X_3) (O=120°C X_1, 20% X_2, 16% X_3), X_1=Barrel temperature, X_2=Feed moisture content, X_3=Amount of cowpea added to rice.

3.2 Effect of extrusion on functional properties

In Table 4, the experimental results obtained for expansion index, bulk density, water absorption and solubility indices, dispersibility and viscosity and the predicted values by the second-order polynomial equation are listed. EI ranged between 124.7 – 133.5, while BD, WAC, WSI, DISP, and AV varies between 0.016 – 0.316g/cm^3, 6.13 – 7.27, 8.36 – 8.94, 98.29 – 99.32 and 22.81 – 25.21Nsm^{-2} respectively. The highest EI (133.5) was from the formulation with 20% moisture and 16% cowpea content and extruded at 120°C, while the least (124.7) was in run containing 25% moisture and 8% cowpea extruded at 100°C (Table 4). This result infers that EI increases with temperature and feed cowpea content, but decreased with increasing moisture. At temperature above 100°C and low feed moisture, materials in extruder tend to be more viscous thereby favouring expansion at the die. Chang and El-Dash (2003) reported that at low moisture level followed by barrel temperature of 120 – 200°C, there is an increased expansion of starch based materials during extrusion. The effect of the processing variables on the BD result indicated significant ($p \le 0.05$) variation among treatments ranging between 0.016gcm^{-3} and 0.316gcm^{-3}. At lower barrel temperature of 100°C, 15% moisture and 24% cowpea levels, the BD was highest and at 140°C, 25% moisture and 8% cowpea composition, the lowest result was recorded. It could be concluded from these results that increasing extrusion temperature negatively influences BD in rice-cowpea extruded samples, while increasing moisture content from 15 to 25% reduces BD and increasing cowpea composition from 8 to 24% improves BD (Table 4). High bulk density in common weaning porridges and

breakfast cereals in Africa has been attributed as one of the major causes of infant malnutrition, since it limits nutrient intake. Low BD observed in these gruels is an indication of its suitability for complementary feeding and breakfast.

The WAI varied between 6.13 and 7.27 in experimental runs 5 and 7 respectively. The barrel temperature and feed composition seems not to significantly ($p \leq 0.05$) affect WAI, but feed moisture content adversely affects the water absorption characteristics. There was also a narrow difference between extrudates in terms of WSI with samples extruded at 100^{o}C barrel temperature, 25% moisture and 24% cowpea having the highest value (8.94), while sample extruded at 140^{o}C, 15% moisture and 24% cowpea recorded the lowest value. This implies that though cowpea composition did not significantly ($p \leq 0.05$) have an impact on the solubility index, increasing temperature from 100^{o}C to 140^{o}C and reduction of cowpea from 15 to 25% causes reduction in WSI. WAI and WSI are important quality parameters often used to define the suitability of the extruded cereal for gruel based products. High WSI is related to stickiness of extruded products (Hashimoto and Grossmann, 2003), and also indicative of degree of degradation of starch molecular components in water and therefore measures the extent of starch conversion to simple molecules during extrusion cooking process which is the amount of soluble polysaccharides released from the starch components after extrusion (Yang *et al*., 2008), while WAI measures the volume occupied by the extrudate particles after swelling in access water and often used as an indication for the integrity of starch in aqueous dispersions such as water and milk (Yang *et al.,* 2008). The produced gruels therefore are of high WAI and WSI which indicate swelling and smooth-sticky gruel.

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Table 4: Observed experimental data and predicted values for extruded rice-cowpea in a twin-screw extruder

Runs	Independent variables			EI		BD		WAI		WSI		DISP		AV	
	BRT	FMC	FBC	OBS	PRED	OBS	PRED	OBS	PRED	OBS	PRED	OBS	PRED	OBS	PRED
1	100	15	8	127.8	127.9	0.090	0.099	6.45	6.49	8.38	8.39	98.65	98.58	25.2	25.3
2	140	15	8	131.1	131.0	0.037	0.035	7.03	7.03	8.77	8.70	98.76	98.79	22.9	22.9
3	100	25	8	124.7	125.4	0.022	0.021	6.52	6.52	8.36	8.36	99.22	99.23	23.9	24.0
4	140	25	8	130.9	130.6	0.016	0.110	6.18	6.17	8.67	8.68	98.84	98.73	23.7	23.4
5	100	15	24	130.7	130.5	0.316	0.300	6.13	6.12	8.70	8.70	98.38	98.59	24.2	24.1
6	140	15	24	129.2	129.3	0.083	0.091	6.64	6.62	8.36	8.37	98.98	98.97	23.8	23.8
7	100	25	24	131.7	131.7	0.080	0.089	7.27	7.27	8.94	8.95	98.84	98.84	23.1	23.2
8	140	25	24	132.7	132.7	0.035	0.033	6.87	6.88	8.62	8.62	98.47	98.50	24.6	24.7
9	86.4	20	16	127.2	127.5	0.142	0.145	6.73	6.74	8.44	8.43	98.93	98.91	23.9	23.9
10	153.6	20	16	131.2	130.9	0.059	0.044	6.85	6.87	8.42	8.43	98.80	98.82	23.3	23.1
11	120	11.6	16	128.9	129.1	0.149	0.153	6.39	6.41	8.73	8.73	99.05	99.03	24.4	24.5
12	120	28.4	16	129.5	129.8	0.053	0.039	6.66	6.67	8.93	8.93	99.17	99.18	24.0	24.2
13	120	20	2.6	129.8	129.6	0.056	0.043	6.37	6.38	8.41	8.42	98.40	98.43	24.1	24.0
14	120	20	29.5	132.8	133.5	0.144	0.147	6.67	6.67	8.63	8.63	98.29	98.25	24.2	24.2
15	120	20	16	133.5	133.0	0.060	0.062	6.28	6.28	8.58	8.58	98.63	98.62	24.7	24.7
Minimum	NA	NA	NA	124.7	125.4	0.016	0.210	6.13	6.12	8.36	8.36	98.29	98.25	22.81	22.90
Maximum	NA	NA	NA	133.5	133.5	0.316	0.300	7.27	7.28	8.94	8.95	99.32	99.23	25.21	25.30
Mean	NA	NA	NA	130.11	130.17	0.089	0.094	6.60	6.61	8.60	8.59	98.76	98.77	24.0	24.0

Regression equation from which the predicted values are determined $Y = \beta_o + \beta_1X_1 + \beta_2X_2 + \beta_3X_3 + \beta_{11}X^2{}_1 + \beta_{22}X^2{}_2 + \beta_{33}X^2{}_3 + \beta_{12} X_1X_2 + \beta_{13} X_1X_3 + \beta_{23} X_2X_3 + \varepsilon$
EI = Expansion index, DB = Bulk density, WAI = water absorption index, WSI = water solubility index, DISP = dispersibility, AV = Apparent viscosity

Dispersibility measures the ability of pulverized extrudates to be wet without the formation of lumps, with simultaneous disintegration of agglomerates. The lowest value was observed in formulation extruded at 120^{o}C, 20% moisture and cowpea composition of 2.6%, while the highest was recorded in run 3 (100^{o}C, 25% moisture and 8% cowpea composition) (Table 4). It is clear therefore from these results that increasing extrusion temperature from 100 to 120^{o}C reduces dispersibility, while increasing feed moisture and cowpea composition favours increased dispersion. The viscosity was highest (13.40 Nsm^{-2}) in sample extruded at 120^{o}C, 20% and 16% moisture and cowpea composition respectively. At 100^{o}C, 25% and 8%, barrel temperature, feed moisture and cowpea composition respectively, the viscosity of the extrudates was lowest (12.51Nsm^{-2}), indicating narrow variations.

3.3 Fitting predictive models for response variables

Predictive models (Equations 15 – 20) were obtained as results of fitting the second order polynomial of Equation (6) to experimental data (Table 4) of the effects of different combinations of extrusion variables on the six response variables. Model fitting is the first step in the optimization of process conditions using RSM and therefore is a key step to obtain the optimal processing parameters. A good fitted regression model therefore is based on the rational experimental design and its corresponding results. If a significant model is established, a quantitative relationship between the independent and response variables would be established that could be used in the prediction of response variables under natural operating conditions. The regression analysis coefficients, ANOVA and lack-of-fit test results are presented in Table 5. The model developed can then be used to establish the optimal process parameters. In this models (Eq. 15 – 20), the coefficient with one factor (X_1, X_2 and X_3) represent the effects of single factor, while coefficient with two of the factors (X_1X_2, X_1X_3, X_2X_3) and those with second-order terms (X_1^2, X_2^2, X_3^2) represent interaction between the three factors and the quadratic effects respectively. A positive sign in front of the terms indicates a synergistic effect, while negative sign indicates an antagonistic effect. The adequacy and fitness of the fitted models were tested by its coefficient of determinations (R^2), lack-of-fit test, p-value, and analysis of residuals. In this study, R^2 value close to unity was considered a better empirical model fit, while no significant lack-of-fit was considered desirable. According to Myers and Montgomery (2002), a satisfactory predictive model should have an adjusted $R^2 \geq 0.80$, a significance level of $p < 0.05$ and lack of fit test >0.1; all these parameters could be used to decide the satisfaction of the models. The predictive models for the relationship between the extrusion variables (X_1, X_2 and X_3) and responses in terms of EI, BD, WAI, WSI, DISP and VISC are outlined in Eqns. 15 to 20. From the results (Table 5 and Eqn. 15-20), the R^2_{adj} were 94.70%, 88.00%, 99.70%, 99.10%, 95.90% and 83.00% respectively for EI, BD, WAI, WSI, DISP and VISC respectively, while the lack-of-fit test were 0.608, 0.485, 0.513, 0.505, 0.297 and 0.200 respectively. R^2_{adj} >82% and lack-of-fit >0.1 are indications that the models could be satisfactorily used to navigate the domain of the experiment. The R^2 measures the proportion of the total variation in response variables which can be explained by the relation between the response variables and the predictor variables. R^2_{adj} close to R^2, Trinh and Kang (2010) reported ensures a satisfactory adjustment of the quadratic models to the experimental data. Hence the regression models therefore could explain the variation in measured properties and could be used to optimize the process variables for optimum product quality.

In pulverized food products, the texture characteristics are strongly related to the bulk density and the ease with which the food material is rehydrated. Food materials are 'instantised' when the surface of each particle is easily wetted then the materials are hydrated and particles sink below the surface of the solvent to disperse rapidly through the solvent. These characteristics are termed wettability, sinkability or dispersibility. For a food material therefore to be considered instant, it should complete these stages within few seconds (Charunuch *et al*., 2011). Since one of the important aims of this study was to produce instant gruel with optimum dispersibility, having model with high R^2, non-significant lack-of-fit and significant model coefficient has fulfilled the achievement of the set objectives.

Table 5: Regression equation coefficients, analysis of variance and lack-of-fit test for response variables in extruded rice-cowpea foods

Terms	Factors					
	EI	BD	WAI	WSI	DISP	VISC
Constant	59.807**	1.1886*	10.714**	6.192**	99.492**	25.5034**
Linear						
X_1	0.8701**	-0.0124*	-0.0627**	0.0485**	-0.021	0.1067
X_2	1.0459**	-0.0588**	0.0280**	-0.1534**	-0.008	-0.3922
X_3	0.7270**	0.04179**	-0.1630**	0.1061**	0.064*	-0.3093*
Square						
X_1^2	-0.0034**	0.00003	0.0005**	-0.0001**	0.0002**	-0.0011*
X_2^2	-0.0503**	0.00049	0.0036**	0.0034**	0.0067**	-0.0054
X_3^2	-0.0080**	0.00018	0.0014**	-0.0003**	-0.0016**	-0.0034*
Interaction						
X_{12}	0.0051**	0.00038**	-0.0022**	0.0000	-0.0018**	0.0046**
X_{13}	-0.0067**	-0.00023**	-0.0001*	0.0010**	0.0002*	0.0031**
X_{23}	0.0238**	-0.00083**	0.0069**	0.0017**	-0.0025**	0.0023
R^2	96.30	91.70	99.80	99.40	97.20	88.30
R^2_{adj}	94.70	88.00	99.70	99.10	95.90	83.00
Lack-of-fit	0.608	0.485	0.513	0.505	0.297	0.200
Model	*	*	*	*	*	*

EI = Expansion index, BD = Bulk density, WAI = Water absorption index, WSI = Water solubility index, DISP = Dispersibility, VISC = Viscosity, * significance at 5%, ** significance at 1%. Regression equation from which the coefficients are determined $Y = \beta_o + \beta_1X_1 + \beta_2X_2 + \beta_3X_3 + \beta_{11}X^2{}_1 + \beta_{22}X^2{}_2 + \beta_{33}X^2{}_3 + \beta_{12}X_1X_2 + \beta_{13}X_1X_3 + \beta_{23}X_2X_3 + \varepsilon$

$$EI = +59.807 + 0.8701X_1 + 1.0549X_2 + 0.7270X_3 - 0.0034X^2{}_1 - 0.0503X^2{}_2 - 0.008X^2{}_3 + 0.0051X_1X_2 - 0.0067X_1X_3 + 0.0238X_2X_3 \quad (R^2 = 0.963) \tag{15}$$

$$BD\ (g/cm^3) = +1.189 - 0.0124X_1 - 0.0588X_2 + 0.0418X_3 + 0.00003X^2{}_1 + 0.00049X^2{}_2 + 0.00018X^2{}_3 + 0.00038X_1X_2 - 0.00023X_1X_3 - 0.00083X_2X_3 \quad (R^2 = 0.917) \tag{16}$$

$$WAI = +10.714 - 0.0627X_1 + 0.0280X_2 - 0.1630X_3 + 0.0005X^2{}_1 + 0.0036X^2{}_2 + 0.0014X^2{}_3 - 0.0022X_1X_2 - 0.0001X_1X_3 + 0.0069X_2X_3 \quad (R^2 = 0.998) \tag{17}$$

$$WSI = +6.192 + 0.0485X_1 - 0.1534X_2 + 0.1061X_3 - 0.00014X^2{}_1 + 0.0034X^2{}_2 - 0.00033X^2{}_3 - 0.001X_1X_3 + 0.0017X_2X_3 \quad (R^2 = 0.994) \tag{18}$$

$$DISP\ (\%) = +99.492 - 0.0208X_1 - 0.0077X_2 + 0.0643X_3 + 0.0002X^2{}_1 + 0.0067X^2{}_2 - 0.0016X^2{}_3 - 0.0018\ X_1X_2 + 0.0002\ X_1X_3 - 0025\ X_2X_3\ (R^2 = 0.972) \quad (19)$$

$$VISC\ (Nsm^{-2}) = +25.503 + 0.1067X_1 - 0.3922X_2 - 0.3093X_3 - 0.0011X^2{}_1 - 0.0054X^2{}_2 - 0.0034X^2{}_3 + 0.0046X_1X_2 + 0.0031\ X_1X_3 + 0.0023\ X_2X_3\ (R^2 = 0.883) \quad (20)$$

3.4 Process and product optimization

Predictive model of each one of the response variables (Equtions15-20) were used to obtain individual desirability level which were utilized for calculating a global desirability (D). The common maximum values for all the response variables were obtained at a D = 0.907, as a result of the best combination of extrusion process variables for the extrusion of the rice-cowpea were barrel temperature 120^{o}C, feed moisture content 20% and 24% cowpea composition. The desirability value obtained was higher than the value considered acceptable (0.6<D<0.08) according to De la Vara and Dominguez (2002). At this optimal process conditions and desirability level, the optimum EI = 126.14, BD = 0.214g/cm^3, WAI = 6.83, WSI = 8.46, DISP = 99.02 and VISC = 12.83Nm^2.

4. Conclusion

In this study, three extrusion cooking variables (barrel temperature X_1, feed moisture content X_2 and rice-cowpea composition X_3) and their effects on extruded food intended to be used as instant breakfast gruel was studied. We developed new equations to study the effects of these variables on the EI and BD of the extrudates and WAI, WSI, DISP and viscosity of the mixed extrudate gruel. The models were fitted to represent the linear, square and interactive relationships between the independent variables and the dependent variables, with significant coefficients, high coefficient of determination and non significant lack-of-fit test. Based on the results of these studies, it was concluded that the independent variables significantly ($p \leq 0.05$) affects the dependent variables and the optimum gruel properties was achieved at 120^{o}C barrel temperature, 20% feed initial moisture content and 24% cowpea in rice flour. The predicted values of the dependent variables were close to experimental values indicating suitability of the model to predict the conditions in real situations. The application of RSM and CCRD in the twin-screw extrusion cooking of broken rice fractions blended with cowpea therefore resulted in extruded products with acceptable gruel characteristics indicating appropriateness of this method for optimizing process conditions for rice-cowpea flour extrusion conditions. It is possible to use the optimal conditions in normal industrial applications. The best combination of extrusion temperature, feed moisture and cowpea levels for the production of extrudates when mixed with water to elaborate gruel with high expansion at the die, low bulk, satisfactory water absorption and solubility indices and viscosity was attained within the experiment domain. Furthermore, the extruded foods have satisfactory EI and BD and other functional qualities and can be used for breakfast cereals and weaning gruel.

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